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(54) Miniaturized reaction apparatus

(57) A stacked plate chemical reactor in which simple plates, each incorporating no surface features other that an opening, are stacked together. When openings in adjacent plates are properly aligned, a fluid pathway is defined between inlet ports for each chemical reactant and an outlet port for a chemical product. In one embodiment of the invention, sixteen simple plates are stacked to provide a reactor incorporating three heat transfer fluid pathways, two reactant fluid pathways, one product fluid pathway, multiple mixing chambers, multiple reaction chambers, two reactant pretreatment heat exchangers, two reaction chamber heat exchangers, and multiple temperature sensor pathways. Precise dimensional control of the reactant fluid pathway height enables stacked laminar flow paths for the reactants to be achieved, allowing efficient and rapid diffusion mixing to occur. Because the simple plates incorporate no features other than openings, fabrication of such plates is easily achieved. Different reactor designs, having additional reactant pathways, more or fewer heat transfer fluid pathways, more or fewer heat exchangers, more or fewer mixing chambers, more or fewer reaction chambers, and more or fewer sensor pathways can readily be achieved by adding or removing plates from the stack, and/or by changing the pattern and number of openings in the simple plates that are used. The simple plates can be held in the stack during use of the chemical reactor using pressure exerted on opposite outer simple plates of the stack, or can be permanently joined. A preferred material for the fabrication of the plates is stainless steel, although other materials such as glass, plastic, and other metals can alternatively be used, which are compatible with the selected reactants and the desired product.

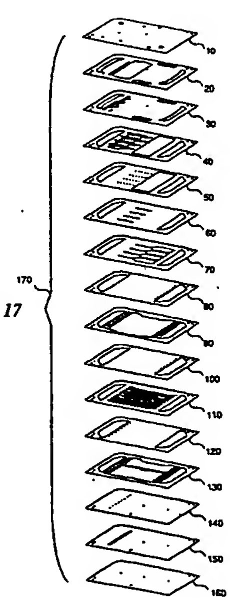

FIG. 17

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production to industrial production, it will be apparent that a microreactor suitable for use in carrying out a variety of chemical processes and having an efficient and low-cost design will be in high demand.

[0009] Several different designs for microreactors have been investigated. For example, such reactors are described in U.S. Patent No. 5,534,328 and U.S. Patent No. 5,690,763 (both listing Ashmead as the inventor). These patents describe reactors structures for chemical manufacturing and production, fabricated from a plurality of interconnected layers. Generally, each layer has at least one channel or groove formed in it and most include orifices that serve to fluidly connect one layer to another. These layers are preferably made from silicon wafers, because silicon is relatively inert to the chemicals that may be processed in the reactor, and because the techniques required to mass produce silicon wafers that have had the required channels and other features etched into their surfaces are well known.

[0010] A disadvantage of the reactors described by Ashmead stems from the rather expensive and complicated process for manufacturing the devices. While silicon wafer technology is advanced to the state that wafers having desired surface features can readily be mass produced, the equipment required is capital intensive, and unless unit production is extremely high, the substantial costs are difficult to offset. While Ashmead does suggest that other materials can be used to fabricate the layers, such as metal, glass, or plastic, the surface features required (grooves, channels, etc.) must still be formed in the selected material. The particular surface features taught by Ashmead require significant manufacturing steps to fabricate. For instance, while forming an opening into a material is relatively easy, forming a groove or channel that penetrates only part way through the material comprising a layer is more difficult, as the manufacturing process must not only control the size of the surface feature, but the depth, as well. When forming an opening that completely penetrates through a material comprising a layer, depth control does not need to be so precisely controlled. Ashmead teaches that not only openings that completely penetrate the layers are required, but also that surface features (grooves/channels) that do not completely penetrate the individual layers are required. Hence, multiple processing steps are required in the fabrication of each layer, regardless of the material selected. Accordingly, it would be desirable to develop a microreactor comprising layers that do not require such detailed fabrication.

[0011] A patent issued to Bard (U.S. Patent No. 5,580,523) describes a modular microreactor that includes a series of fluidly connected modules, each module having a particular function (fluid flow handling and control, mixing, chemical processing, chemical separation, etc.). Bard specifically teaches that the plurality of modules are mounted laterally on a support structure, and not stacked, as disclosed by Ashmead. In a preferred embodiment of Bard, silicon wafer technology is again used to etch channels and/or other features into the surface of a silicon wafer. Other disclosed fabrication techniques include injection molding, casting, and micromachining of metals and semiconductor substrates. Again, the processing required to fabricate the individual modules goes beyond merely forming a plurality of openings into each component. Furthermore, the lateral layout of the reactor described by Bard requires a larger footprint than a stacked plate reactor. In Bard's reactor, the more modules added, the larger the footprint of the entire reactor. In contrast, when additional plates are added to a stacked plate reactor, the footprint of the reactor does not change, which can be a distinct advantage, as in many work environments the area an apparatus occupies on a work bench or floor is more valuable than the vertical height of the apparatus. It would be desirable to provide a reactor design that has a minimal footprint, while still providing the flexibility to add components to customize the reactor for a particular process.

[0012] In U.S. Patent No. 5,961,932 (Ghosh), a reactor is described that is formed from a plurality of ceramic layers, which are fluidly connected, at least one layer including a permeable partition. In particular, Ghosh describes the desirability of sizing fluid channels appropriately to provide for laminar flow and mixing via diffusion, rather than mixing via turbulence. In his preferred embodiment, Ghosh describes that channels, chambers, and passageways are formed in each layer. The particular process Ghosh describes to accomplish this task involves fabricating the layers from "green" or uncured ceramic, which once shaped as desired, must be sintered. Significantly, the sintering process changes the size of the ceramic layer, so that the sizes of the features formed into the ceramic layer in the initial stages of production are not the sizes of the features in the finished product. It would be desirable to provide a reactor design in which the dimensions of the individual components can be rigidly controlled during fabrication, and not subject to shrinkage, which can negatively effect the dimensions of the finished reactor. This object is particularly important when a reactor design focuses on achieving a laminar flow, because precise dimensional control of fluid pathways in the reactor must be maintained to achieve a consistent laminar flow.

[0013] In all of these prior art reactors, relatively complicated manufacturing techniques are required. The manufacture of layers of silicon material requires a large capital investment. Sintering of a ceramic material requires the precise control of the shrinkage process, or individual components of a desired size cannot be achieved. In all cases, the prior art teaches that complicated structures (for example, fluid channels and reaction chambers) must be etched or otherwise fabricated in each layer. Additionally, orifices or passages also need to be formed in each layer, so that fluids can move between adjacent layers of the reactor. Thus, a series of different manufacturing steps typically must be performed for each layer. It would be desirable to provide a reactor design offering the advantages described above, that is relatively simple to manufacture, so as to minimize capital investment in scaling up production from the laboratory to the industrial level. It is therefore an aim of this invention to provide a micro-scale reaction apparatus that can be

force. In such an embodiment, a housing provides the compressive force, producing a pressure acting on the outer simple plates. The mean surface roughness of the plates should be less than about 1 micrometers, and the simple plates should be substantially free of scratches. The pressure should be greater than or equal to 50 Newtons per square centimeter. In another embodiment, the simple plates are permanently joined. When permanently joined, the mean surface roughness of the plates is preferably less than about 5 micrometers. Permanent joining can be achieved using diffusion welding or vacuum soldering.

[0027] Preferably, when the thickness of the intermediate simple plates that are adjacent to a heat exchanger is about 0.3 millimeters. When a series of openings in the simple plates of the chemical reactor defines a fluid path for a heat transfer fluid that flow through more than one heat exchanger, the flow rate and fluid pressure of the heat transfer fluid within each such heat exchanger are substantially.

[0028] Another aspect of the present invention is directed to a method for producing stacked plate reactor, which includes steps generally consistent with the apparatus described above.

Brief Description of the Drawing Figures

[0029] The foregoing aspects and many of the attendant advantages of this invention will become more readily appreciated as the same becomes better understood by reference to the following detailed description, when taken in conjunction with the accompanying drawings, wherein:

FIGURE 1 is a plan view of a top simple plate of a preferred embodiment for a chemical reactor in accord with the present invention, including openings for reactants, heat transfer media inlets and outlets, and an opening for a temperature sensor;

FIGURE 2 is a plan view of the second simple plate of the preferred reactor, showing a plurality of heat exchanger manifolds, a first reactant opening, a second reactant distributor, and a first heat exchanger;

FIGURE 3 is a plan view of the third simple plate of the preferred reactor, illustrating a plurality of heat exchanger manifolds, a first reactant distributor, and a second reactant opening;

FIGURE 4 is a plan view of the fourth simple plate of the preferred reactor, showing two heat exchanger manifolds, an inter-digital-mixer for the reactants, and a second heat exchanger;

FIGURE 5 is a plan view of the fifth simple plate of the preferred reactor, showing two heat exchanger manifolds, a plurality of openings for the reactants, and a second heat exchanger;

FIGURE 6 is a plan view of the sixth simple plate of the preferred reactor, showing two heat exchanger manifolds, and a plurality of reactant fluid channels;

FIGURE 7 is a plan view of the seventh simple plate of the preferred reactor, showing two heat exchanger manifolds, a plurality of reactant fluid channels, and a plurality of mixing chambers for the reactants;

FIGURE 8 is a plan view of the eighth simple plate of the preferred reactor, showing two heat exchanger manifolds and a plurality of product openings;

FIGURE 9 is a plan view of the ninth simple plate of the preferred reactor, showing two heat exchanger manifolds, a third heat exchanger, and a plurality of product openings;

FIGURE 10 is a plan view of the tenth simple plate of the preferred reactor, showing two heat exchanger manifolds and a plurality of product openings;

FIGURE 11 is a plan view of the eleventh simple plate of the preferred reactor, showing two heat exchanger manifolds, and a plurality of reaction channels;

FIGURE 12 is a plan view of the twelfth simple plate of the preferred reactor, illustrating two heat exchanger manifolds, and a plurality of product openings;

FIGURE 13 is a plan view of the thirteenth simple plate of the preferred reactor, illustrating two heat exchanger manifolds, a plurality of product openings, a plurality of temperature sensor openings, and a fourth heat exchangers that is separated into an upper section and a lower section;

FIGURE 14 is a plan view of the fourteenth simple plate of the preferred reactor, illustrating a plurality of product openings and a plurality of temperature sensor openings;

FIGURE 15 is a plan view of the fifteenth simple plate of the preferred reactor, illustrating a product channel and a plurality of temperature sensing openings;

FIGURE 16 is a plan view of the sixteenth and bottom simple plate of the preferred reactor, illustrating a product withdrawal opening and a plurality of temperature sensor openings;

FIGURE 17 is an exploded isometric view of the preferred reactor, illustrating how all sixteen simple plates are stacked;

FIGURE 18A is an exploded isometric view of the first six simple plates of the preferred reactor, illustrating a fluid path for a first reactant;

FIGURE 18B is an exploded isometric view of the first six simple plates of the preferred reactor, illustrating a fluid

reactants necessitates an appropriate material be selected for fabricating the simple plates of the reactor.

[0034] A preferred embodiment of the present invention, as described below, represents a design that has been optimized for a liquid/liquid phase reaction involving two reactants. It should be understood that the underlying concept of the present invention, i.e., a reactor formed of a stack of plates incorporating only openings, can be applied to many other types of reactions, such as liquid/gas, gas/gas, liquid/solid, or gas/solid. As will be described in detail below, the preferred embodiment includes four heat exchangers; three heat transfer media pathways, and two reactant fluid pathways. However, it should similarly be understood that similar stacked plate reactors can be easily designed to include more or fewer heat exchangers, more or fewer heat transfer media pathways, and more reactant pathways.

[0035] The disclosed preferred reactor has been optimized for processing two component liquid/liquid reactions that generally require only temperature controls. However, it should be understood that other types of reactions, requiring additional processing controls, can be processed in a stacked simple plate reactor in accord with the present invention, if the reactor is optimized for that control parameter. For example, reactors can readily be designed to incorporate magnetic, piezoresistive, piezoelectric, shape memory, radioactive, catalytic, and electrostatic control parameters.

[0036] The plurality of stacked simple plates enables a reactor to be constructed that performs from one to all of the following functions: reactant conditioning, control of reactant supply, thermal pre-treatment, combination and mixing of reactants under controlled thermal conditions, intermediate thermal treatment, post-procedural isothermal containment, post-procedural thermal treatment of reactant products, and product separation. In particular, simple plates can readily be designed and fabricated in which the dimensional characteristics of the reactant fluid passages formed by the interconnected openings of the simple plates provide for a stacked laminar flow of the reactants. Such a stacked laminar flow ensures that a particularly efficient type of mixing, referred to as diffusion mixing, can occur.

[0037] The quality of the interconnections between the simple plates is of great importance, since the interconnections must be free of gaseous and liquid leakage. This requirement is achieved through a combination of specially prepared surfaces and use of simple plates that are fabricated to close tolerances. The individual simple plates can be assembled by pressure fitting (using clamps or a housing that encloses the simple plates and applies a compressive force to the outer plates), or individual simple plates can be permanently assembled using diffusion welding technology, vacuum soldering, or other suitable techniques for joining the simple plates together.

[0038] The pressure fitting technique has the advantage of allowing a reactor to be built using specific simple plates that can readily be disassembled so that the reactor design can be changed by adding or removing simple plates. In this manner, the same simple plates can be used in more than one reactor to effect different chemical processes. However, if the simple plates are assembled using .pressure fitting, very good control of the surface finishes is required, with almost no scratches on the surface of the simple plates, and a mean surface roughness less than 1 µm. The pressure that should be applied to maintain a stack of simple plates that have been fabricated from metals into a reactor, to prevent gas or liquid leakage, is preferably about 50 Newtons/cm^2.

[0039] Successful diffusion welding to join metallic simple plates also requires a substantially scratch free surface, although the mean surface roughness can be increased up to 5 µm. In diffusion welding, the simple plates are pressed together and heated to about 1000° C in a vacuum or inert atmosphere. At such temperatures, ions from each surface diffuse across the surface boundary layers, thus joining the surfaces.

[0040] Vacuum soldering is a technique that requires a mean surface roughness of less than 5 µm, although more scratches can be tolerated than in diffusion welding. The simple plates are first coated with a thin film (3-5 µm) of silver, either by sputtering, vapor deposition, or electrical deposition. Other metallic films, such as gold or copper, can also be used. The simple plates are then heated in a vacuum to about 900° C. The silver liquefies, filling any voids due to scratches or surface irregularities, and bonds the simple plates together to form a reactor.

[0041] It should be noted that when the reactor is assembled using diffusion welding or vacuum soldering, a superior bond can be obtained by minimizing the surface area that is to be bonded. Thus, simple plates that incorporate one or more openings occupying a significant portion of the surface area of the simple plates can be more efficiently bonded with either of these two techniques than simple plates with few or small openings that comprise only a small portion of their total area.

[0042] Preferably, any stacked simple plate reactor should have the ability to maintain a desired narrow temperature range within the reactor, so that reaction dynamics can be closely controlled. In a preferred embodiment, the reactant and heat transfer media enter the stacked simple plate reactor via vertically oriented fluidic channels. Reacted product and spent heat transfer media exit the reactor via similarly disposed vertically oriented fluidic channels. The chemical processing operations occur in horizontally disposed channels within the reactor. It should be noted that the use of the term channel when used in conjunction with a stacked simple plate reactor should not be construed to mean that such a channel corresponds to a groove formed into the surface of an individual plate. While each individual simple plate only has openings and no grooves, channels or other fluid pathways are easily obtained in a stacked simple plate reactor. To form a channel, an elongate narrow opening is formed in one simple plate and that simple plate is sandwiched between two simple plates that do not have a corresponding elongate opening. The top of the channel is defined by the upper simple plate, the sides of the channel are defined by the sides of the opening formed in the middle simple

differently. Heat transfer media A, entering the reactor from inlet 12a in simple plate 10, passes through second simple plate 20 via a heat transfer media A intake manifold 22a. After progressing through subsequent simple plates of the preferred reactor, heat transfer media A again passes through simple plate 20, this time via a heat transfer media A exhaust manifold 22b. From that point, heat transfer media A exits the preferred reactor via outlet 12b in top simple plate 10.

[0050] In second simple plate 20, heat transfer media B services a first heat exchanger 24. Heat transfer media B enters first heat exchanger 24 through inlet 14a in top simple plate 10, and flows from first heat exchanger 24 via outlet 14b in top simple plate 10. Reactant A flows into a Reactant A distributor 25 in second simple plate 20, while Reactant B, passes through second simple plate 20 via Reactant B opening 17. Heat transfer media C flows through second simple plate 20 using a heat transfer media C intake manifold 26a, passing through subsequent simple plates and then returning via a heat transfer media C exhaust manifold 26b. Temperature sensor A (not shown) passes through second simple plate 20 within temperature sensor opening 19.

[0051] The fluid paths of heat transfer media A-C, and the purposes of the four separate heat exchangers in the preferred stacked simple plate reactor will be discussed in detail below, with respect to FIGURES 19A, 19B, and 19C. Briefly, the purpose of first heat exchanger 24 in simple plate 20 is to modify the temperature of Reactants A and B before they are combined. In many reactions, it is desirable for the reactants to be at the same temperature prior to mixing the reactants. However, there are certain reactions for which it is beneficial for a Reactant A and a Reactant B to be brought to different temperatures prior to mixing. It is envisioned that a stacked simple plate reactor can be designed to achieve this goal by changing the openings through the simple plates described above to provide for an additional heat exchanger. Thus, separate heat exchangers can be provided and used to separately modify the temperatures of Reactants A and B.

[0052] FIGURE 3 provides details of the passages in the third layer of the preferred reactor. A third simple plate 30 includes chamfer 11. Heat transfer media A flows through third simple plate 30 via heat transfer media A intake manifold 22a and out through heat transfer media A exhaust manifold 22b. Note that heat transfer media B does not flow through the third layer of the preferred reactor, as heat transfer media B feeds first heat exchanger 24 in second simple plate 20 in the second layer of the preferred reactor and is then exhausted through outlet 14b in top simple plate 10. Heat transfer media C flows through the third layer via heat transfer media C intake manifold 26a, and also via heat transfer media C exhaust manifold 26b. Reactant A flows through third simple plate 30 using a plurality of Reactant A fluid openings 35, which are fed from Reactant A distributor 25 in second simple plate 20. Reactant B enters the third layer of the preferred reactor via Reactant B opening 17 of second simple plate 20, and then flows into a Reactant B distributor 37 in third simple plate 30. Third simple plate 30 also includes temperature sensor opening 19.

[0053] FIGURE 4 provides details of the fourth layer of the preferred reactor, showing a fourth simple plate 40, which has chamfer 11. Heat transfer media A flows through fourth simple plate 40 via a heat transfer media A'manifold 42a and out through a heat transfer media A exhaust manifold 42b. It should be noted that the shape of the heat transfer media A intake manifold and exhaust manifold in simple plate 40 have changed in shape from the corresponding heat transfer media A intakes 22a and exhaust manifolds 22b in second simple plate 20 and third simple plate 30. The functional significance of this shape change is to reduce the overall pressure drop in the reactor. The slight curve on the outside edges of intake manifold 42a and exhaust manifold 42b has been included to optimize the fluid flow of heat transfer media A within the preferred reactor. It should also be noted that because intake manifold 42a and exhaust manifold 42b are larger in size than intake manifold 22a and exhaust manifold 22b, the surface area of the simple plate is reduced. As noted above, a smaller surface area results in a superior bond if diffusion welding or vacuum soldering is used to assemble the simple plates in the stack.

[0054] Fourth simple plate 40 of FIGURE 4 incorporates a second heat exchanger 46, which is serviced via heat transfer media C intake manifold 26a and heat transfer media C exhaust manifold 26b of simple plate 30. Second heat exchanger 46 moderates the temperatures of Reactants A and B as the two reactants enter mixing chambers in a subsequent layer of the preferred reactor.

[0055] Fourth simple plate 40 incorporates important features that effect the fluid paths of Reactants A and Reactants B. Collectively, these features are referred to as an "inter-digital-mixer." The purpose of the inter-digital-mixer is to precisely align the fluid paths of Reactants A and B, such that a stacked laminar flow is enabled, while also ensuring that an equal pressure drop is achieved for the two reactants. A stacked laminar flow is preferred to enable diffusion mixing to occur. Diffusion mixing is recognized as being both extremely fast and efficient.

[0056] Reactant A flows through a series of openings 45, while Reactant B flows through a series of openings 47. Further details of the preferred stacked laminar flow will be provided below in conjunction with FIGURES 20-23. A series of dashed lines has been included on FIGURE 4, to illustrate that certain portions of openings 45 and 47 are aligned. As will become clear in examining the details of the fifth layer of the preferred reactor and a simple plate 50, as illustrated in FIGURE 5, once Reactants A and B have passed through the fluid paths formed by openings 45 and 47, the fluid paths of the reactants will be aligned. Simple plate 40 also includes temperature sensor opening 19.

[0057] Referring now to FIGURE 5, a fifth simple plate 50 includes chamfer 11. Heat transfer media A passes through

plate 90 via mixed reactant openings 85. The path of heat transfer media A through ninth simple plate 90 is relatively complex, as compared to the paths of heat transfer media A through the previous layers of the preferred reactor. Heat transfer media A flows through ninth simple plate 90 via an intake manifold 42a, and an exhaust manifold 42b. Heat transfer media A is also flowing through third heat exchanger 93, which has a series of cutouts on both the right and left edges of the heat exchanger. These cutouts are disposed such that they overlap the enlarged areas of heat transfer mediaA intake and exhaust manifolds 82a and 82b of eighth simple plate 80. In this manner, heat transfer media A intake manifold 82a of eighth simple plate 80 services both heat transfer media A intake manifold 42a of ninth simple plate 90, and also heat exchanger 93 of ninth simple plate 90. Heat exchanger 93 is used to modify the temperature of a plurality of reaction channels in an eleventh layer, as will be described more in detail below, in conjunction with FIGURES 10 and 11.

[0065] It should be noted that the shape of third heat exchanger 93 has been designed and empirically tested to maximize fluid flow and heat transfer. For instance, the cut outs on the right side of third heat exchanger 93 are required for mixed reactant openings 85 to be included in layer 9. The left side of third heat exchanger 93 similarly has cut outs, but no mixed reactant openings 85 are located on the left side of simple plate 90, and theoretically cut outs are not required. However, as will be seen in FIGURE 13, a fourth heat exchanger is required to have cut outs on the left side for product openings to pass through layer 13. Because the third and fourth heat exchangers moderate the temperature of the same area, reaction channels in layer 11, the fluid dynamics of both heat exchangers should be as similar as possible. Thus, while each heat exchanger is required to have cut outs on only one of the right or left side for fluid openings, each heat exchanger has been designed with cut outs on both the right and left sides to achieve as much fluidic equilibrium as possible.

[0066] The indentation on the top edge of third heat exchanger 93, and a similarly shaped protrusion on the bottom edge of third heat exchanger 93, are included so that the flow of heat transfer media within third heat exchanger 93 matches as closely as possible the flows of reactants/product through reaction channels in layer 11. The shapes of the reaction channels and the third heat exchanger have been designed to enable other openings to exist on the simple plates (such as intake and exhaust manifolds, and other required fluid passages) and to match the fluid paths of the heat transfer media to the reactants fluid paths, as closely as possible. Compare the shape of third heat exchanger 93 to the reaction channels of layer 11, and the similarity will be apparent. If the upper and lower indentation and protrusion where not included in third heat exchanger 93, then the flow of heat transfer media through the third heat exchanger would generally flow from the cut outs on the right to the cut outs on the left, with little fluid flowing between these parallel flows. As a result, the heat transfer media in third heat exchanger 93 would have a flow pattern that does not match the flow pattern of mixed Reactants A and B in the reaction channels of layer 11, thus reducing the effectiveness of the third heat exchanger.

[0067] FIGURE 10 provides details of a tenth layer of the preferred reactor. FIGURE 10 shows a tenth simple plate 100 having chamfer 11. Heat transfer media A flows through the tenth layer using a heat transfer media A intake manifold 82a, and a heat transfer media A exhaust manifold 82b. Note again that the size of the intake and exhaust manifolds for heat transfer media A in the tenth layer have changed, now matching the size of the heat transfer media A intake and exhaust manifolds of the eighth layer. However, in the eighth layer, the purpose of the size change in the heat transfer media A intake and exhaust manifolds was to feed heat exchanger 93 in the ninth layer. The reason for the change in size of the heat exchanger intake and exhaust manifolds in the tenth layer is not related to servicing a heat exchanger. As noted above, the less surface area a simple plate has, the stronger the bond between the simple plates. The enlarged openings reduce the surface area, thus improving the bond strength. Another benefit is that, as will become apparent as later Figures are examined, tenth simple plate 100 (with the exception of the location of chamfer 11) is a mirror image of a subsequent simple plate 120 in the twelfth layer. Simple plate 120 is required to have the larger heat transfer media A intake and exhaust manifolds to feed a fourth heat exchanger in the thirteenth layer immediately below. Merely by changing the location of chamfer 11, the same fabrication configuration can be used to manufacture simple plate 100 and simple plate 120.

[0068] It should be noted that third heat exchanger 93 of the ninth layer of the preferred reactor modifies the temperature of a solid portion of tenth simple plate 100. As will be seen in examining an eleventh simple plate 110 of the eleventh layer of the preferred reactor, that solid portion corresponds to a plurality of reaction channels in eleventh simple plate 110. Because such a large portion of tenth simple plate 100 is required to be solid to provide for a heat transfer surface, the use of larger heat transfer media A intake and exhaust manifolds is important to reduce the surface area of simple plate 100, to increase the bond strength. The mixture of Reactants A and B flows through tenth simple plate 100 via a plurality of mixed reactant openings 85.

[0069] FIGURE 11 provides details on the eleventh layer of the preferred reactor, showing eleventh simple plate 110, with chamfer 11. Heat transfer media A flows through eleventh simple plate 110 via heat transfer media A intake manifold 42a, and heat transfer media A exhaust manifold 42b. The mixed Reactants A and B enter a plurality of reaction channels 115 from the right side of plate 110. Reaction channels 115 are fed from the plurality of mixed reactant openings 85, which form a passage from mixing chambers 77 of the seventh layer, through aligned mixed reactant openings 85

modify the temperature of a solid portion of tenth simple plate 100 that forms the upper surface of reaction channels 115. Fourth heat exchangers 133a and 133b moderate the temperature of a solid portion of twelfth simple plate 120 that forms the lower surface of reaction channels 115. It should be noted that the heat exchangers of the preferred reactor actually moderate the temperature of a solid portion of simple plates both above and below the opening that corresponds to the heat exchanger, that the purpose of the heat exchangers is to control the temperature of the reacting product in reaction channels 115. Note that fourth heat exchangers 133a and 133b are moderating the temperature of a solid portion of both simple plate 120 of layèr 12 and a simple plate 140 of layer 14. While moderating a solid portion of simple plate 120 does effect the temperature of the product in reaction channels 115, the moderation of the solid portion of simple plate 140 serves no functional purpose. In the preferred reactor, the modification of the temperature of non-target portions of simple plates, such as simple plate 140, does not cause any problems. However, it is envisioned that in different stacked plate reactors, such non-target temperature changes could be undesirable. In such reactors, a simple plate that does not conduct thermal energy (i.e. whose thickness is sufficient to prevent heat transfer) could be used to isolate the heat exchangers to avoid non-target temperature changes.

[0077] The fourteenth layer of the preferred reactor is a relatively simple layer, involving only product fluid openings to withdraw reacted product from the reactor, and passages for temperature sensors as described above. FIGURE 14 illustrates fourteenth simple plate 140, which includes chamfer 11. Temperature sensor passages 139a, 139b, and 139c enable temperature sensors to be inserted into the reactor to monitor the temperature of reaction channels 115 as discussed above. Product openings 125 are used to direct the reacted product to lower levels of the reactor.

[0078] The fifteenth layer is also relatively simple. FIGURE 15 illustrates a fifteenth simple plate 150, having chamfer 11. The plurality of product openings 125 from the fourteenth layer are combined into a single product channel 155. Again, three temperature sensor openings 139a, 139b, and 139c are included to enable temperature sensors to be passed deeper into the core of the reactor.

[0079] The final layer of the preferred reactor is the sixteenth layer. FIGURE 16 illustrates a sixteenth simple plate 160, also having chamfer 11. The single product channel 155 of the fifteenth layer is reduced in area to a single product outlet port 165. Again, temperature sensor openings 139a-139c are available so that temperature sensors can be inserted deeper into the reactor. It should be noted that in the preferred reactor the thickness of both the top simple plate 10 and the bottom simple plate 16 are significantly greater than the thickness of the intermediate simple plates. The greater thickness provides both greater structural integrity, as well as helping to thermally isolate the inner layers of the reactor from the outside environment.

[0080] FIGURE 17 is an exploded isometric view of a preferred reactor 170 that includes the sixteen layers described in regard to FIGURES 1-16. Simple plates 10-160 are shown stacked in order so that the relative positions of each simple plate to each other may be examined. The preferred dimensional thickness of each simple plate is as follows:

Top simple plate 10	3.0 mm.
Second simple plate 20	0.3 mm.
Third simple plate 30	0.3 mm.
Fourth simple plate 40	0.3 mm.
Fifth simple plate 50	0.3 mm.
Sixth simple plate 60	0.3 mm
Seventh simple plate 70	0.2 mm.
Eighth simple plate 80	0.3 mm.
Ninth simple plate 90	0.6 mm
Tenth simple plate 100	0.3 mm.
Eleventh simple plate 110	0.2 mm.
Twelfth simple plate 120	0.3 mm
Thirteenth simple plate 130	0.6 mm.
Fourteenth simple plate 140	0.3 mm.
Fifteenth simple plate 150	0.3 mm
Sixteenth simple plate 160	3.0 mm

[0081] Simple plates 10 and 160 (the top and bottom simple plates) are thicker than other plates to provide greater structural stability. Simple plates 20-60, 100, 120, 140 and 150 are much thinner, to enhance heat transfer. As will be discussed below, a thickness of 0.3 mm provides a reasonable heat transfer ability for a wide variety of materials. Simple plate 70 is thinner by 1/3 to ensure proper laminar flow within mixing chambers 75. Simple plate 11 is the same thickness as simple pate 70, to maintain fluidic equilibrium conditions in the reactor. Simple plates 90 and 130 are thicker than other plates to provide a larger mass of fluid in the heat exchangers 93, 133a and 133b. It should be noted

heat exchanger 24 via outlet port 14b in top simple plate 10. The purpose of heat-exchanger 24 is to adjust the temperature of the solid section of portion of the third layer that is immediately above the inter-digital-mixer (openings 45 and 47) in fourth simple plate 40. In this manner, heat exchanger 24 is moderating the temperatures of Reactants A and B prior to the reactants being mixed together. It is contemplated that for the majority of reactions; it will be desirable for Reactants A and B to be of similar temperature. Those of ordinary skill in the art will readily understand, however, that there may be some reactions in which Reactant A and Reactant B will preferably be kept at separate temperatures. It is contemplated that a different stacked plate design using the same principles of the invention can be designed and fabricated to provide for a separate heat exchanger to individually modify the temperatures of Reactants A and B.

[0088] FIGURE 19B illustrates the fluid path that heat transfer media C takes through layers 1-4 of the preferred reactor. Heat transfer media C enters the reactor through inlet 16a in top simple plate 10 and then proceeds through heat transfer media C intake manifolds 26a on simple plates 20 and 30, in layers 2 and 3, respectively. Heat transfer media C then enters heat exchanger 46 on fourth simple plate 40 of layer 4 and exits heat exchanger 46 by utilizing heat transfer media C exhaust manifolds 26b of simple plates 30 and 20, in layers 3 and 2, respectively. Heat transfer media C then exits the reactor using outlet port 16b of top simple plate 10. The purpose of second heat exchanger 46 is to modify the temperature of the solid portion of sixth simple plate 60 that corresponds to the mixing chambers 77 of seventh simple plate 70. Because the mixing of chemicals often spontaneously generates heat, a great deal of heat can be generated as Reactants A and B are thoroughly mixed. Second heat exchanger 46 is thus able to cool Reactants A and B while in mixing chambers 77, so that the temperatures of the reactants do not exceed the ideal temperature for the desired reaction. Second heat exchanger 46 occupies both the fourth and fifth layers (simple plates 40 and 50), to increase the capacity of the heat exchanger.

[0089] FIGURE 19C illustrates the fluid path for heat transfer media A as it passes through the first thirteen layers of preferred reactor 170. Heat transfer media A enters the reactor at top simple plate 10 via intake port 12a. The heat transfer media A then passes through identical heat transfer media A intake manifolds 22a on simple plates 20 and 30 of layers 2 and 3 respectively. Heat transfer media A continues to pass through heat transfer media A intake manifolds in layers 4, 5, 6 and 7, via intake manifolds 42a. It should be noted that intake manifolds 42a differ in size and shape relative to the intake manifolds 22a of layers 2 and 3. The functional purpose of the size change is both reduce potential pressure drops within the fluid paths of the reactor, as well as to reduce the surface area of simple plates 40-70 to enhance bonding.

[0090] In layer 8, the shape of heat transfer media A intake manifold 82a changes once again. The purpose of the size change between the heat transfer media A intake manifolds in layers 7 and 8 is so that heat transfer media A can be fed into two separate sections of the layer 9. In a first heat transfer media A fluid path in layer 9, heat transfer media A flows into a heat transfer media A intake manifold 42a, and from there to heat transfer media A intake manifold 42a of tenth simple plate 100 in layer 10. From there, heat transfer media A flows to heat transfer media A intake manifold 42a in layer 11, an enlarged heat transfer media A intake manifold in layer 12, and then to heat transfer media A intake manifold 42a in layer 13.

[0091] In a second heat transfer media A fluid path in layer 9, fluid flows out of heat transfer media A intake manifold 82a of eighth simple plate 80 and into third heat exchanger 93 on ninth simple plate 90 of layer 9. As discussed above, the purpose of third heat exchanger 93 is to moderate the temperature of the solid portion of layer 10 immediately adjacent to reaction channels 115 in layer 11. Heat transfer media A exits heat exchanger 93 by returning to layer 8 via heat transfer media A exhaust manifold 82a, which is enlarged and overlaps the right end of third heat exchanger 93.

[0092] Simple plate 100 of layer 10 includes enlarged heat transfer media A intake manifold 82a (as well as exhaust manifold 82b). It should be noted that reaction channels 115 of layer 11 are not quite long enough to overlap the enlarged heat transfer medial intake and exhaust manifolds 82a and 82b, thus no heat transfer media enters reaction channels 115. Here, the functional purpose of the size change of the intake and exhaust manifolds is to reduce the surface area of tenth simple plate 100, to enhance bonding, rather than to feed a heat exchanger (as in layer 8 and eighth simple plate 80).

[0093] Referring now to layer 11, note that again the size and shape of heat transfer media A intake manifold 42a has changed relative to the intake manifolds of layers 8 and 10. This size change relates to maintaining a calculated fluidic equilibrium throughout the micro reactor. However, it is contemplated that the overall effect of the size change is relatively minor, and that an effective micro reactor can be achieved without changing the size of the intake manifolds on layer 11.

[0094] In layer 12, the size and shape of heat transfer media A intake manifold 82a is again enlarged, to once again divert some heat transfer fluid A into a second fluid path that services fourth heat exchangers 133a and 133b of layer 13. Heat transfer media A also flows into a heat transfer media A intake manifold 42a in layer 13. The functional purpose of heat transfer media A intake manifold 42a of layer 13 is to ensure that the fluid pressure within fourth heat exchangers 133a and 133b matches the fluid pressure within third heat exchanger 93. Note both the third and fourth heat exchangers are moderating the temperature of reaction channels 115, and thus preferably both heat exchanges should have similar flow characteristics.

[0102] The height reduction of the combined volume of fluid channels 65/75 to mixing chambers 77 further enforces a stacked laminar flow. Preferably the channel height is reduced by two thirds in the mixing area respective to the lamination channel (fluid channels 65 and 75), resulting in a thickness of each individual fluid layer of less than 50 µm. In this dimension, mixing by diffusion is enforced. In a laminar flow regime, fluids stay together as stacked layers when being forced into a narrower mixing area. To achieve the same volumetric flow rate in the mixing area as in the lamination channel, the mixing area is broader than the lamination channels (fluid channels 65/75), as can be seen in the isometric view of simple plate 70 (see also FIGURE 7).

[0103] It is critically important that when the reactants enter the lamination channels (fluid channels 65/75), they are stacked one on top of the other, rather than side by side. In the preferred reactor, the inter-digital-mixer of layer 4 (fourth simple plate 40) ensures that the plurality of reactant fluid streams are properly aligned so that when they enter the lamination channels (fluid channels 65/75) the reactant streams are properly stacked on top of one another. FIGURE 22 illustrates one design for an inter-digital-mixer that *will not* ensure the reactants will properly stack on one another, while FIGURE 23 illustrates the preferred design of the inter-digital-mixer *that does* enable proper stacking.

[0104] In FIGURE 22, both Reactant A opening 45a and Reactant B opening 47 are similar in size and shape. These openings are shown as superimposed over fluid channel 65. It should be understood that openings 45a and 47 are part of fourth simple plate 40 (the fourth layer), while fluid channel 65 is part of sixth simple plate 60. However, openings 45a and 47 are in fluid communication, and are shown here together to illustrate the fluid path enabled by this poorly designed inter-digital-mixer. In FIGURE 22, an area 186 is indicated by dashed lines. In an enlarged view of area 186, details of the fluid paths enabled by this poorly designed inter-digital-mixer can be seen.

[0105] As noted above, the function of the inter-digital-mixer is to separate two individual reactant fluid paths into a plurality of fluid paths. It is important that these plurality of fluid paths are properly aligned, so that the stacked laminar flow described above can be achieved. Thus, the design of the inter-digital-mixer is extremely important, as a poorly designed inter-digital-mixer will not ensure that the desired stacked laminar flow is achieved.

[0106] In the enlarged detail of area 186, a Reactant A fluid path 145a and a Reactant B fluid path 147 are shown. Note that as illustrated, both the reactants enter fluid channel 65 in a side by side laminar flow pattern, rather than in the desired stacked laminar flow pattern, because the reactants are entering fluid channel 65 from opposing sides, rather than from the same side. The side by side effect is due to how the fluid fronts of each fluid path propagate. Note that each fluid path (145a and 147) is changing direction by approximately 90 degrees as the fluid path enters fluid channel 65. The fluid front of each fluid path will have a tendency to propagate fastest along the inside of that 90 degree corner, as the fluid on the inside of the corner has less distance to travel. When fluid path 145a encounters fluid path 147, each respective fluid front is maximized along the channel wall corresponding to the inside of the 90 degree corner associated with each fluid path. Thus a side by side laminar flow condition results.

[0107] Compare the poorly designed inter-digital-mixer of FIGURE 22 with the preferred inter-digital-mixer of FIGURE 23. The shape of Reactant A opening 45 has been changed to include a curve at the portion of the openings that are superimposed over fluid channel 65. These curves ensure that even during low pressure or low flow conditions, Reactant A enters fluid channel 65 from the same side as Reactant B. FIGURE 23 similarly includes dashed lines indicating an area 187. In the enlarged view of area 187, a Reactant A fluid flow 245 can be seen with a Reactant B fluid flow 247 stacked on top, rather than side by side, as in FIGURE 22. As noted above, in layers 4-7 (the inter-digital-mixer, fluid channels 65 and 75, and mixing chambers 77) the reactants flow from left to right, Reactant A enters fluid channel 65 first, and is thus on the bottom of the channel, with Reactant B stacked on top. Because the openings alternate between Reactant A and Reactant B, a six layer stacked laminar flow is achieved, with three layers of Reactant A and three layers of Reactant B in an alternating pattern. While additional simple plates could have been added to the preferred reactor to define a reactant fluid path that also ensured that the reactants would enter fluid channel 65 from the same side, this modification would not only increase the cost and complexity of the reactor, but it would also result in different pressure drops between the reactants. This difference would result in less than ideal mixing dynamics, and thus, is not a preferred solution. It should be noted that the shape of openings 45, in particular, the curve radius, length, and width have been carefully selected to achieve an equal pressure drop for both Reactants A and B.

Exemplary Chemical Reaction Performed in a Stacked Simple Plate Reactor

[0108] The described chemical reaction belongs to the class of organometallic conversions, i. e., the addition of an organolithium compound to a carbonyl compound. Cyclohexanone (1) reacts in a one step procedure with methyl lithium to produce the 1,2-addition product 1-methyl-cyclohexanol (2).

time chamber can be added to the reactor, either by using additional simple plates, or by adding a separate residence chamber module downstream of the reactor.

[0116] The resultant product stream leaves the reactor via a Teflon™ tube into a collection flask that is filled with 2N hydrochloric acid. Instant quenching of the addition adduct and excess reagent takes place.

Benefits of the Simple Plate Stacked Reactor:

[0117] Advantages of the stacked simple plate reactor system are precise temperature control, exact adjustment of reaction time, and eliminating the need of a protective atmosphere, since the reactor is a closed environment. Enhanced safety is provided due to the small quantities of material, and the closed environment operating conditions.

[0118] The system is especially advantageous when large quantities of product are required, because the reactor can work continuously, and can be operated for hours, even up to days, without maintenance. Accordingly, automated production of large amounts of the desired product without the loss of efficiency and safety can be achieved. Additional product can be obtained by operating additional reactors in parallel under identical operating conditions.

System Description:

[0119] The reactants are provided in conventional laboratory bottles with tube connectors. The bottles are connected to a pump module by Teflon™ tubes. Inside a pump module disposed upstream from the pumps are three way valves, which are connected to the reactants, the solvents and pump inlet. For conditioning the stacked simple plate reactor, the valves are set to the solvents, so that the pumps first fill the whole system with solvent until the stacked simple plate reactor reaches thermal equilibrium. Then the valves are set to the reactants, enabling the pumps to deliver the reactants into the stacked simple plate reactor. A filter is placed inline between the pump outlet and reactor inlet to avoid clogging of the system by particulates. Fluidic connection of pumps and reactor can be achieved by commercially available HPLC fittings. Controlling the temperature of the stacked simple plate reactor is achieved by pumping heat transfer media from a cryostat into the internal heat exchangers of the stacked simple plate reactor. Product coming out of the system is collected in a conventional laboratory bottle.

Measuring and Automation Control Devices:

[0120] All pumps, valves and cryostats are preferably controlled by a microcontroller or computer, programmed with appropriate software, enabling convenient adjustment and control of the system. The following sensor devices are optionally used to provide analog signals that are converted to digital signals for input to the microcontroller or computer, to facilitate more efficient manual or automated control of the chemical process:

- Pressure sensors disposed downstream from each pump and at the inlet and outlets of the stacked simple plate reactor.
- Temperature sensors integrated in the stacked simple plate reactor and disposed close to the mixing zone and at the reactor outlet.
- Optional flow sensors introduced into each reactant stream for improved flow adjustment.

Excellent control and adjustment of flow and ratio of the reactants, determination of the pressure buildup inside the system by differential pressure measurement, and exact adjustment and control of the reaction temperature can thus be achieved.

[0121] Although the present invention has been described in connection with the preferred form of practicing it, those of ordinary skill in the art will understand that many modifications can be made thereto within the scope of the claims that follow. Accordingly, it is not intended that the scope of the invention in any way be limited by the above description, but instead be determined entirely by reference to the claims that follow.

Claims

1. Reactor for contacting and optionally reacting one chemical with at least one other chemical to form a chemical product, said reactor comprising a plurality of simple plates stacked in layers, each simple plate having at least one opening that extends therethrough, an opening in each simple plate overlapping at least one other opening in an adjacent simple plate, thereby forming at least one passage within the reactor to convey and mix said one chemical with said at least one other chemical, the chemical product being formed within said passage by a reaction between said one chemical and said at least one other chemical.

(b) provides a fluid path for one of:

i. said at least two reactants;
ii. the chemical product; and
iii. a heat transfer fluid;

(c) facilitates mounting a temperature sensor;
(d) separates a single fluid path into a plurality of fluid paths;
(e) provides at least one of a heat exchanger, a mixing chamber, an inter-digital-mixer, and a reaction pathway; and
(f) enhances a flow characteristic of a fluid, including a direction of a fluid flow and a pressure drop of the fluid within the chemical reactor.

12. A method for fabricating a chemical reactor for combining at least two reactants to produce a desired product, comprising the steps of:

(a) providing a plurality of simple plates, said plurality of simple plates including two outer simple plates and at least one intermediate simple plate in which is formed at least one opening, at least one of the outer simple plates including inlet ports for said at least two reactants and an outlet port for discharging the desired product;
(b) stacking said plurality of simple plates such that said at least one opening in said at least one intermediate simple plate is in fluid communication the inlet ports and the outlet port; and
(c) securing said plurality of simple plates in the stack.

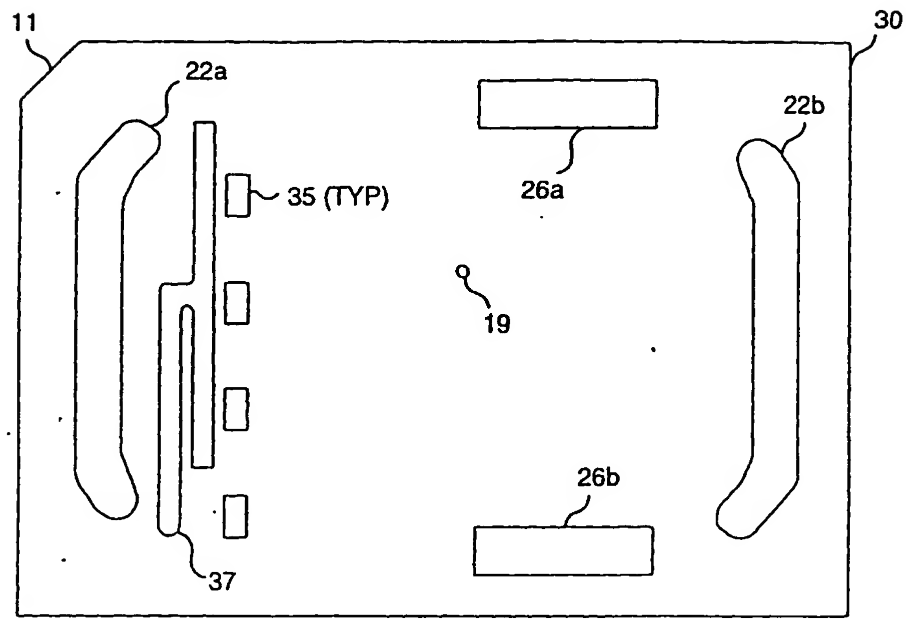


FIG. 3

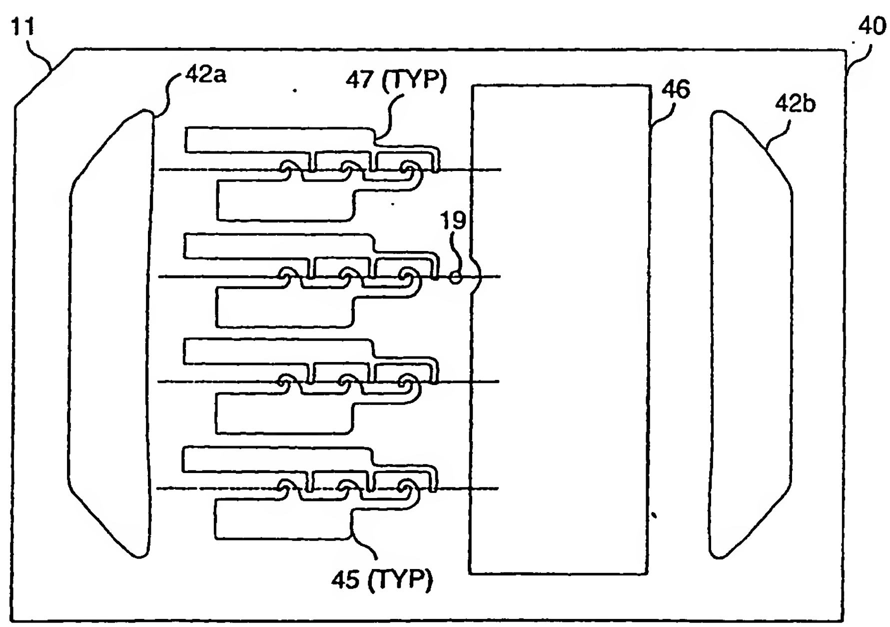


FIG. 4

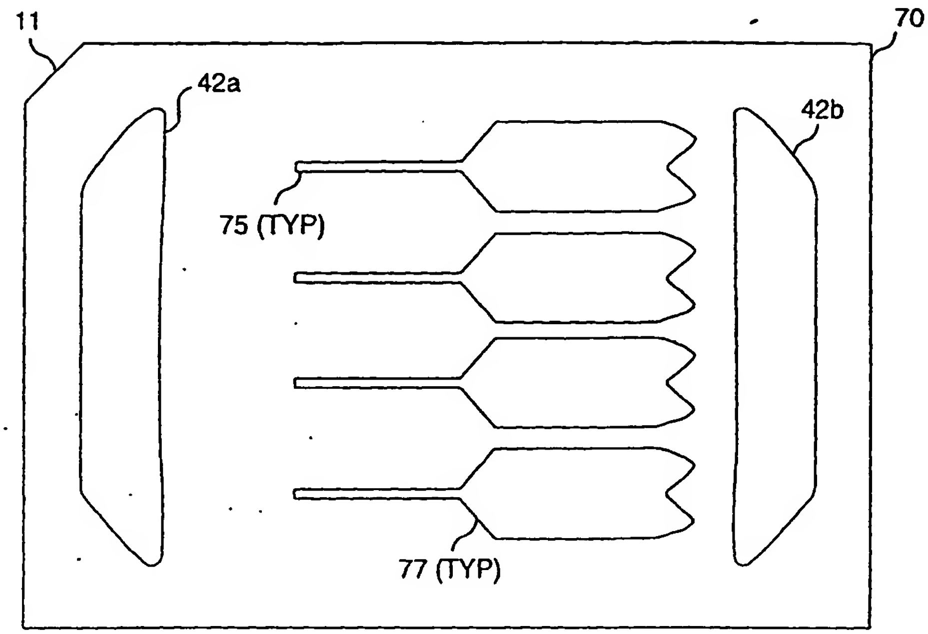


FIG. 7

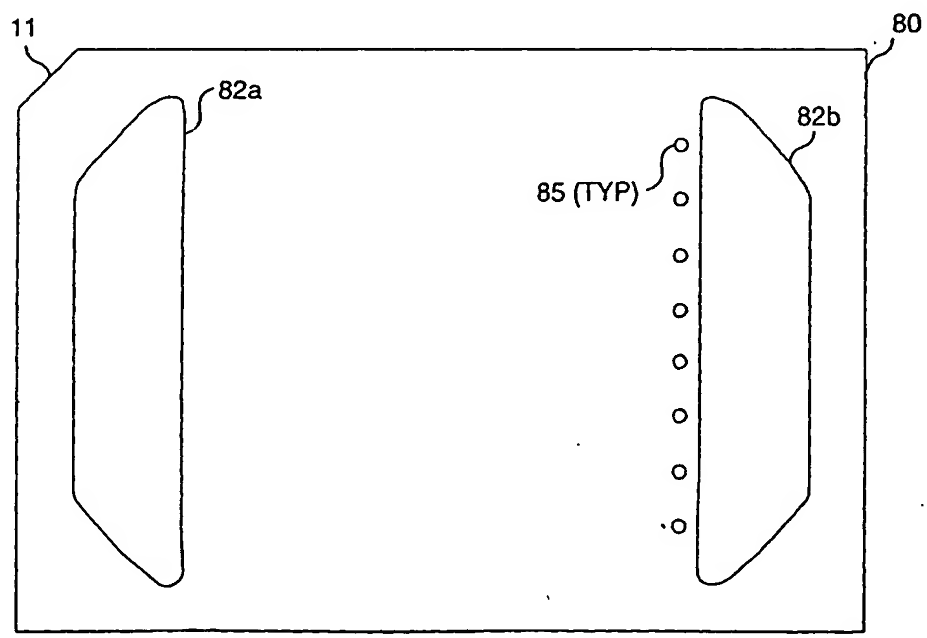


FIG. 8

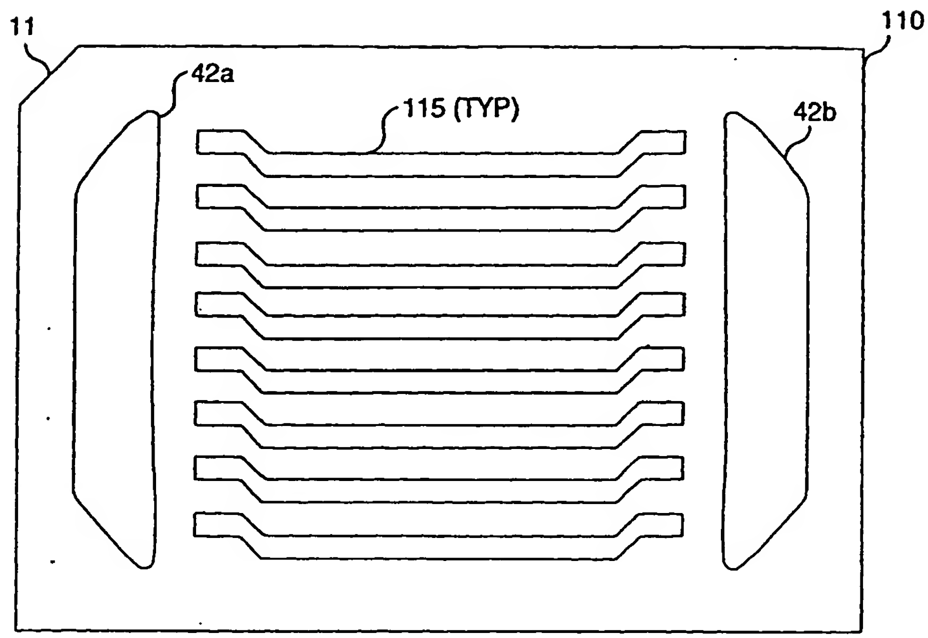


FIG. 11

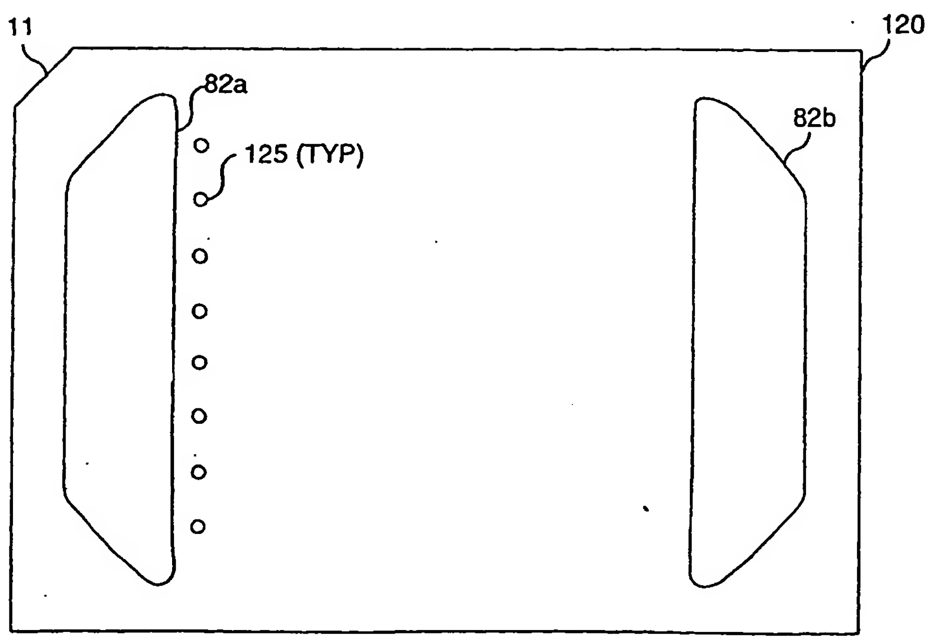


FIG. 12

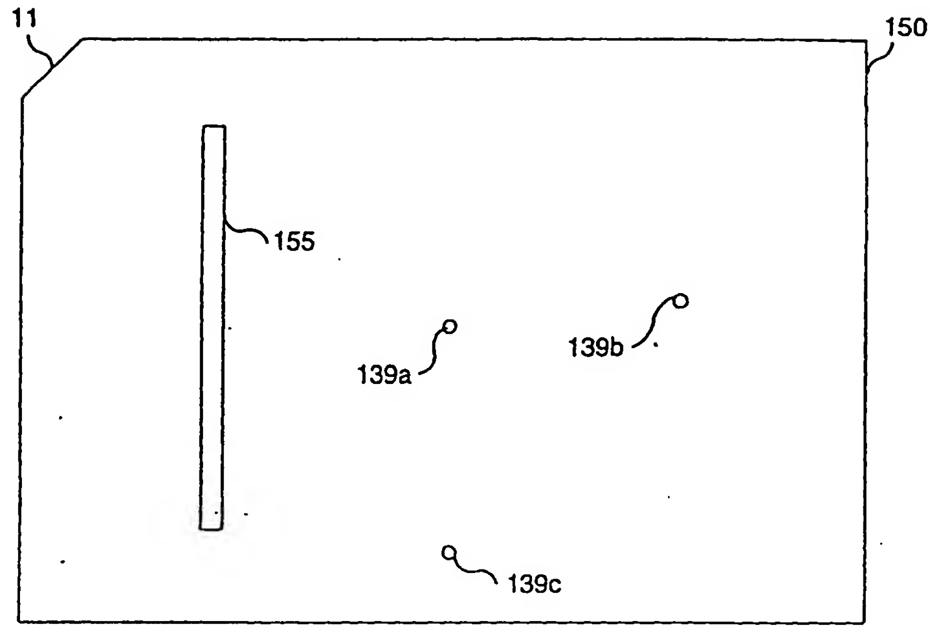


FIG. 15

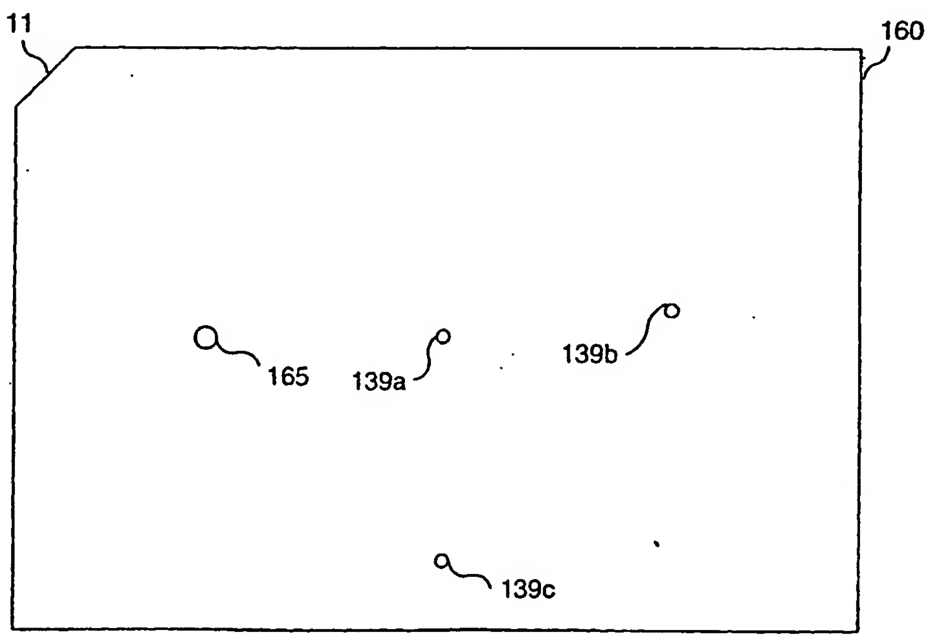


FIG. 16

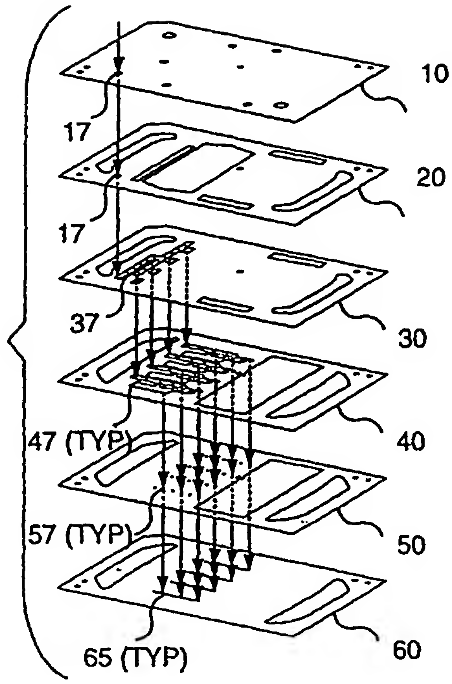


FIG. 18A

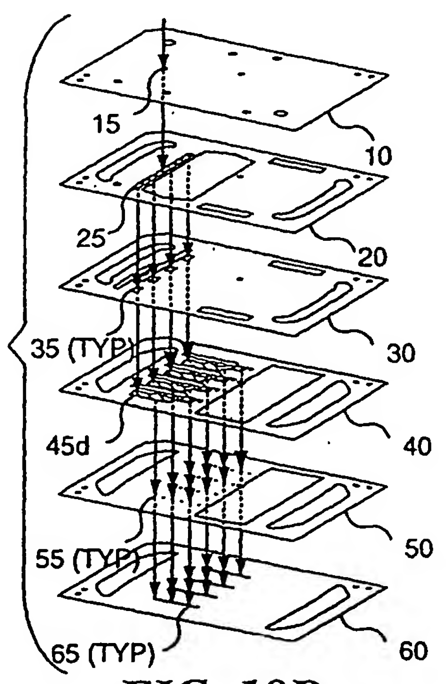


FIG. 18B

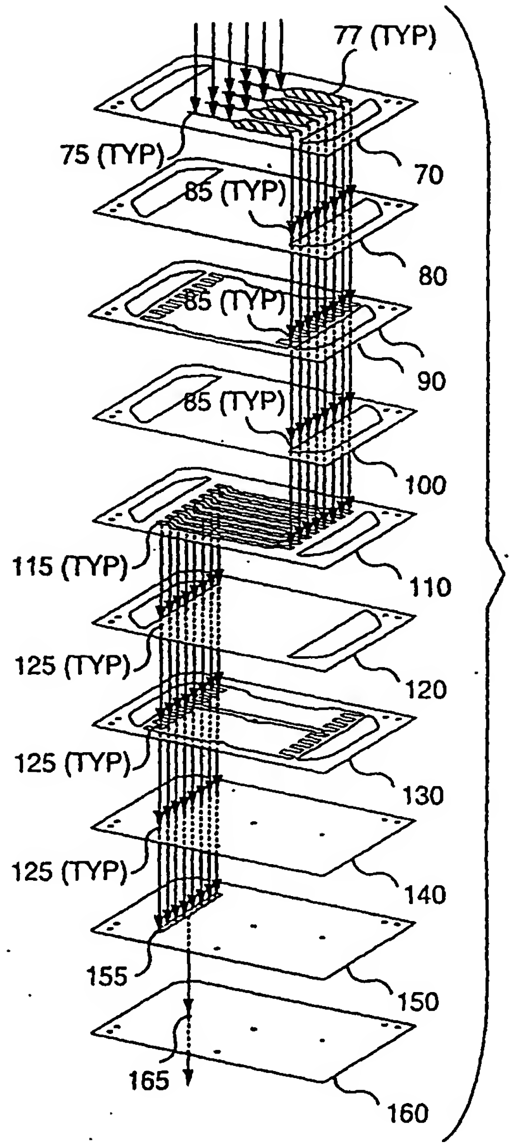
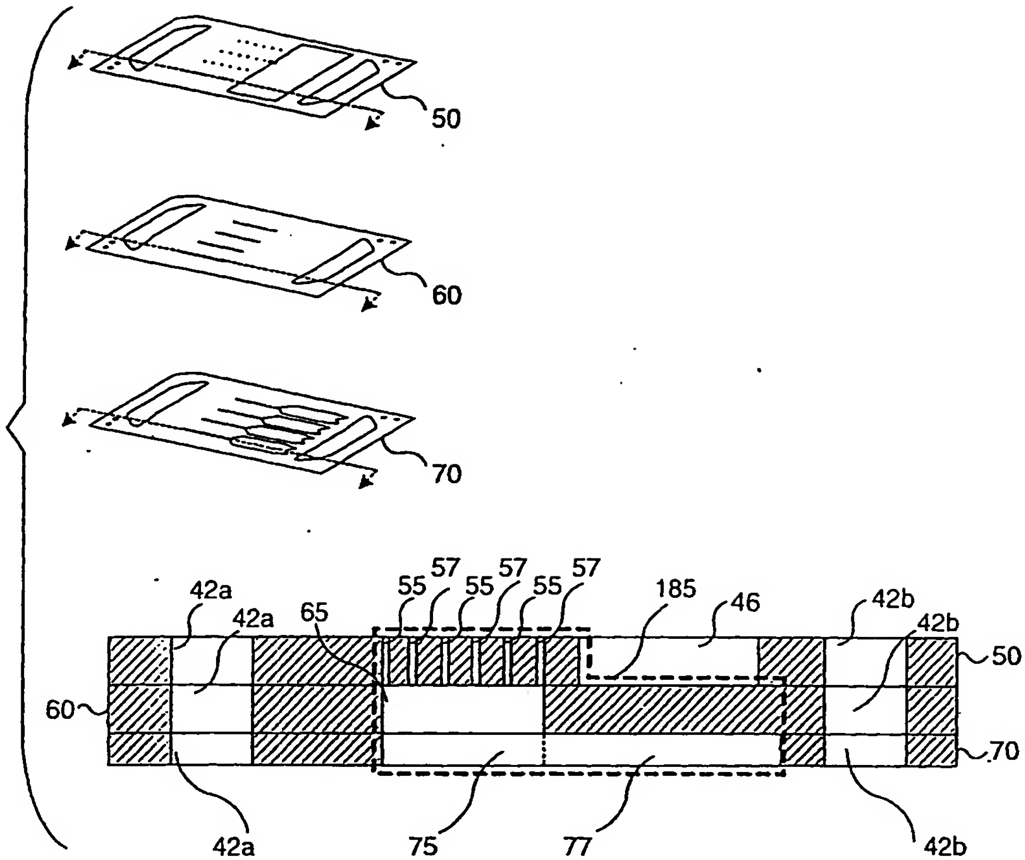


FIG. 18C


50
60
70
42a
42a
65
55
57
55
57
55
57
185
46
42b
42b
50
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70
42a
75
77
42b

FIG. 20

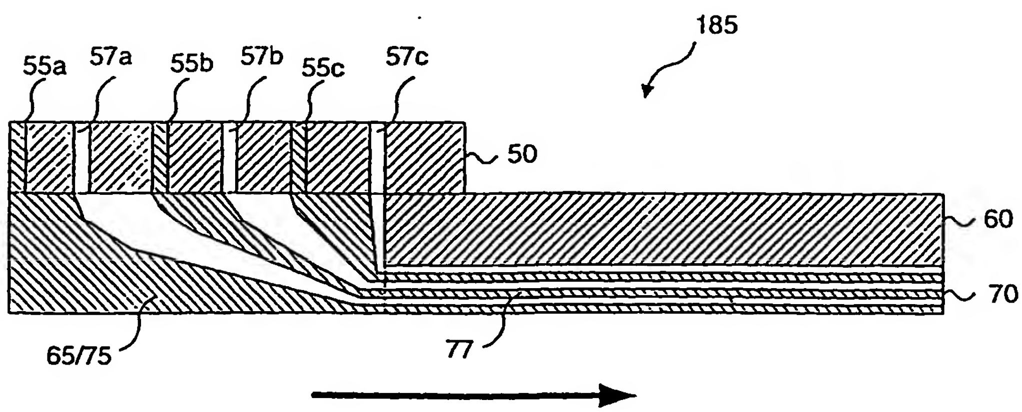
185
55a
57a
55b
57b
55c
57c
50
60
70
65/75
77

FIG. 21